

National Institute for Environmental Studies  
Certificate of Analysis  
NIES CRM No. 10-d Rice Flour-Unpolished

This environmental certified reference material (CRM), which continues NIES CRM No.10 series, was developed and certified by the National Institute for Environmental Studies (NIES) for the determination of cadmium and other elements in unpolished rice and materials of similar matrix. Certified values are given for eight elements and reference values for a further five elements.

**Certified values of NIES CRM No.10-d**

| Element | Mass fraction |                 |   |             | Analytical method               |
|---------|---------------|-----------------|---|-------------|---------------------------------|
|         | Unit          | Certified value | ± | Uncertainty |                                 |
| Mg      | %             | 0.156           | ± | 0.007       | ICP-OES, ICP-MS                 |
| K       | %             | 0.384           | ± | 0.024       | AAS, ICP-OES, ICP-MS            |
| Mn      | mg/kg         | 9.59            | ± | 0.82        | ICP-OES, ICP-MS                 |
| Fe      | mg/kg         | 16.4            | ± | 1.8         | ICP-OES, ICP-MS                 |
| Cu      | mg/kg         | 7.18            | ± | 0.63        | ICP-OES, ICP-MS                 |
| Zn      | mg/kg         | 26.5            | ± | 1.1         | ICP-OES, ICP-MS                 |
| Sr      | mg/kg         | 0.217           | ± | 0.024       | ICP-OES, ICP-MS                 |
| Cd      | mg/kg         | 0.401           | ± | 0.034       | AAS, ICP-OES, ICP-MS, ID-ICP-MS |

All certified values were determined based on dry mass. The uncertainty attached to the certified values is the expanded uncertainty using a coverage factor  $k = 2$ , corresponding to a confidence interval of approximately 95 %.

AAS, atomic absorption spectroscopy; ICP-OES, inductively coupled plasma optical emission spectrometry; ICP-MS, inductively coupled plasma mass spectrometry; ID-ICP-MS, isotope dilution-ICP-MS

**Reference Values of NIES CRM No.10-d**

| Element | Mass Fraction |                 | Analytical method    |
|---------|---------------|-----------------|----------------------|
|         | Unit          | Reference value |                      |
| P       | %             | 0.386           | AAS, ICP-OES         |
| S       | %             | 0.128           | ICP-OES              |
| Na      | mg/kg         | 13.5            | AAS, ICP-OES, ICP-MS |
| Ca      | mg/kg         | 107             | ICP-OES              |
| Mo      | mg/kg         | 0.762           | ICP-OES, ICP-MS      |

All reference values were determined based on dry mass. AAS, atomic absorption spectroscopy; ICP-OES, inductively coupled plasma optical emission spectrometry; ICP-MS, inductively coupled plasma mass spectrometry

## **Characterization**

The property values of the material were statistically determined based on chemical analyses by 8 organizations (including 14 laboratories) using a wide range of methods. A property value satisfying the following conditions was accepted as a certified value: 1) the relative standard deviation associated with the mean of the laboratory means was 5 % or less; 2) the number of laboratories contributing to the mean of the laboratory means was at least eight; 3) the number of analytical methods contributing to the mean of the laboratory means was at least two. The uncertainty attached to the certified values is the expanded uncertainty using a coverage factor  $k = 2$ , corresponding to a confidence interval of approximately 95 %. A property value failing to satisfy one or two of the NIES criteria for certification but supplying valuable additional information about the material is given as a reference value. All certified and reference values were determined based on dry mass.

## **Description of the Material**

The CRM is supplied as fine powder in an amber glass bottle. It is off-white in color.

## **Preparation of the CRM**

This CRM was produced from rice that had been hydroponically cultivated under strictly controlled conditions within the Ecosystem Research Field of NIES. The raw material for the CRM was unpolished rice (about 10 kg) that had been harvested by hand. It was dried at 70 °C for 24 h, crushed in a rotor mill with a 200 µm-sieving ring and homogenized with a V-blender. The powdered unpolished rice was placed in amber glass bottles (15 g in each bottle, 609 bottles), and sterilized by <sup>60</sup>Co irradiation (20 kGy) . All procedures complied with ISO GUIDE 34.

## **Homogeneity**

Mass fractions of multi-elements, including those for which certified values are given, were determined by ICP-OES and ICP-MS in material taken from 10 bottles selected from the total 609 bottles by stratified random sampling. The between-bottle variation evaluated by a one-way analysis of variance (ANOVA) showed the homogeneity standard deviations between bottles for the analytes to be less than 1 %. The material, therefore, is sufficiently homogeneous for its intended use as a reference material.

## **Stability**

Stability tests demonstrated that long-term (6 months) and short-term (2 weeks at -20 °C or 50 °C) variations in the concentrations of all measured elements in the material were insignificant.

## **Instructions for Use**

1. Care should be taken to avoid contamination when opening the bottles. It is desirable to use up the contents as quickly as possible after opening.
2. This CRM should be kept tightly closed in its original bottle and stored in a desiccator at room temperature (Ö30 °C).
3. Prior to weighing portions for analysis, the contents of the bottle should be shaken gently.
4. It is recommended that a sample intake of 0.2 g is the minimum for convenient handling.
5. Precautions should be taken to avoid inhalation of the material.
6. This CRM should not be used for purposes other than research. When disposing of the material, local laws

concerning processing and disposal of waste materials should be strictly adhered to.

7. The mass fractions of elements in this CRM are reported on a dry mass basis. This CRM, as received, has a moisture content of approximately 3 % measured in NIES by drying a separate sub-sample for 4 h at 85 °C. Correction to dry mass should be determined by drying a separate sub-sample at the time of use.

#### **Expiry Date of Certification**

The expiry date for the certified values of this CRM is September 2022 assuming that the recommended storage conditions are adhered to. NIES will notify via its website if any changes in the contents are recognized within the term of validity.

#### **Collaborating Laboratories in Analysis**

The certified and reference values for this CRM were based on analytical values from the following participating organizations:

National Institute for Environmental Studies; Environmental Control Center Co.,Ltd; IDEA Consultants, Inc.; Japan Food Research Laboratories; Murata Measuring Instrument Service Ltd.; Naitoh Environmental Science Co.,Ltd; Nittech Research Corporation; Shimadzu Techno-Research, Inc.

#### **Technical Information**

Technical information and the latest research reports regarding this material can be obtained from the following address.

#### **Correspondence should be addressed to:**

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September 15, 2012

Takashi Imamura

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